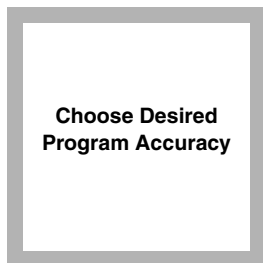


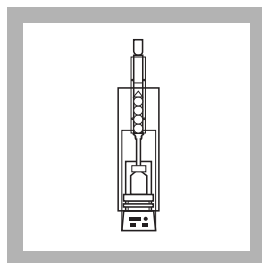
NITROGEN, TOTAL KJELDAHL (0 to 150 mg/L)

Nessler Method* (digestion required)

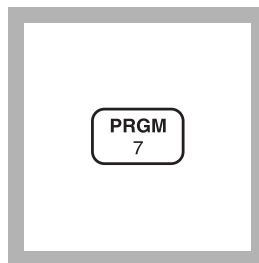
For water, wastewater and sludge



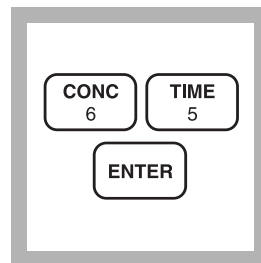
1. A User-Entered Calibration is necessary to obtain the most accurate results. See the User Calibration section following these steps.



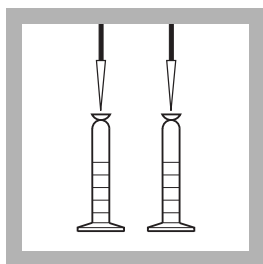
2. Digest the sample as described in the Digesdahl Apparatus Instruction manual. Digest an equal amount of deionized water as the blank.



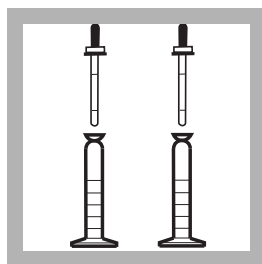
3. Enter the stored program number for total Kjeldahl nitrogen. Press: **PRGM**
The display will show:
PRGM ?



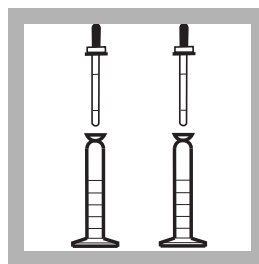
4. Press: **65 ENTER**
The display will show **mg/L, TKN** and the **ZERO** icon.



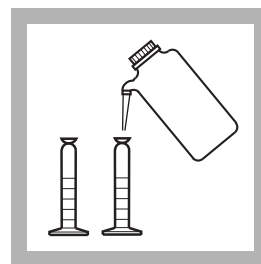
5. Select the appropriate analysis volume of the digested sample given in *Table 1* on page 355. Pipet the analysis volume from the sample and the digested blank into separate 25-mL mixing graduated cylinders.



6. Add one drop of TKN Indicator to each cylinder. Add 8.0 N KOH dropwise to each cylinder, mixing after each addition. Continue until the first apparent blue color is visible.



7. Add 1.0 N KOH to each cylinder, one drop at a time, mixing after each addition. Continue until the first permanent blue color appears.

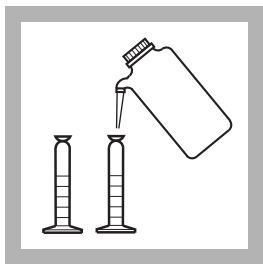


8. Fill both mixing cylinders to the 20-mL mark with deionized water. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix. Add 3 drops of Polyvinyl Alcohol Dispersing Agent to each cylinder. Invert several times to mix.

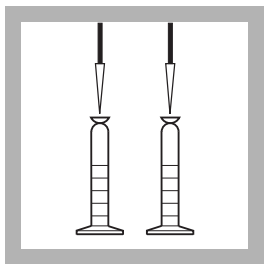
Note: Hold the dropping bottles upright while dispensing.

* Adapted from: Hach et al., *Journal of Association of Official Analytical Chemists*, 70 (5) 783-787 (1987); Hach et al., *Journal of Agricultural and Food Chemistry*, 33 (6) 1117-1123 (1985); *Standard Methods for the Examination of Water and Wastewater*.

NITROGEN, TOTAL KJELDAHL, continued

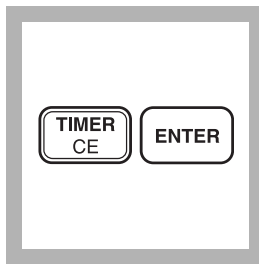


9. Fill both cylinders to the 25-mL mark with deionized water.

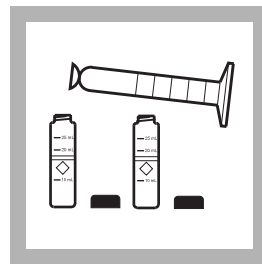


10. Pipet 1 mL of Nessler's Reagent to each cylinder. Stopper, invert repeatedly. The solution should not be hazy.

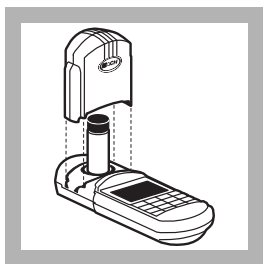
Note: Any haze (turbidity) will cause incorrect results.



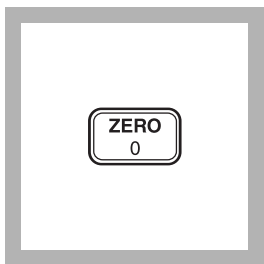
11. Press:
TIMER ENTER
A two-minute reaction period will begin.



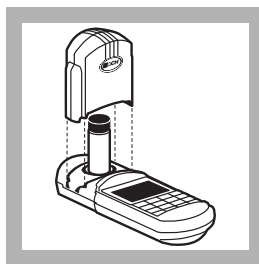
12. When the timer beeps, pour the contents of each cylinder into a separate labeled sample cell.



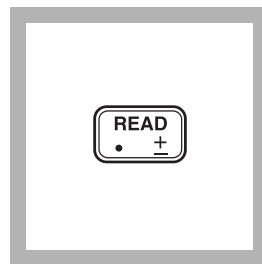
13. Place the blank into a cell holder. Tightly cover the sample cell with the instrument cap.



14. Press: **ZERO**
The cursor will move to the right, then the display will show:
0. mg/L TKN



15. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



16. Press: **READ**
The cursor will move to the right, then the result in mg/L total Kjeldahl nitrogen will be displayed.

Note: Standard Adjust may be performed using a prepared ammonia standard (see Standard Adjust in Section 1).

NITROGEN, TOTAL KJELDAHL, continued

$$\text{ppm TKN} = \frac{75 \times A}{B \times C}$$

17. Use the formula shown to calculate the final TKN value.

Where:

A = mg/L displayed

B = g (or mL of water) sample taken for digest

C = mL analysis volume of digested sample (step 5).

Note: For water samples ppm TKN = mg/L TKN.

Note: For maximum accuracy, the reagent blank value may be determined by repeating procedure using reagents only.

Subtract the reagent blank value from the reading on the display.

Table 1 Analysis Volumes Based on Concentration

AQUEOUS SAMPLES (Solutions of suspensions in water- less than 1% solids)	
Expected Nitrogen Concentration (mg/L)	Analysis Volume (mL)
0.5-28	10.00
2-112	5.00
11-560	2.00
45-2250	1.00
425-22500	0.50
DRY SAMPLES	
Expected Nitrogen Concentration (mg/L)	Analysis Volume (mL)
42-2200	10.0
106-5600	5.00
350-18000	2.00
1000-56000	1.00
4200-220000	0.50
OILS AND FATS	
Expected Nitrogen Concentration (mg/L)	Analysis Volume (mL)
85-4500	10.0
210-11000	5.00
2100-11000	1.00

NITROGEN, TOTAL KJELDAHL, continued

Sampling and Storage

Collect samples in a cleaned glass or plastic container. Adjust the pH to 2 or less with sulfuric acid (about 2 mL per liter) and cool to 4 °C. Preserved samples can be stored up to 28 days.

Accuracy Check

Kjeldahl Nitrogen Standard Method

This procedure checks digestion efficiency and indicates that amount of bound nitrogen that is freed during digestion. The methods and standards available to check digestion technique are found in the Accuracy Check section following the procedures in the Digesdahl Digestion Apparatus Instruction Manual. Using the digested Kjeldahl standard, perform the above TKN analysis on the colorimeter. The TKN value should come within about $\pm 3\%$ of the value of the prepared Kjeldahl standard.

Standard Solution Method (to check calibration accuracy only)

Add one drop of TKN Indicator to each of two 25-mL graduated mixing cylinders. Fill one cylinder to the 20-mL mark with deionized water. Fill the other cylinder to the 20-mL mark with a 1.0 mg/L Ammonia Nitrogen Solution. Add 3 drops of Mineral Stabilizer to each cylinder. Invert several times to mix. Add 3 drops of Polyvinyl Alcohol Dispersing agent to each cylinder. Perform the TKN procedure as described in Steps 9 to 16. This display should show 26-27 mg/L TKN.

User Calibration

For most accurate results, use a user-calibrated program. The Standard Adjust feature should not be used with a user-entered calibration; it will hinder performance.

A one-time setup of a program for TKN is recommended for each new lot of reagents. A new calibration may be performed for each lot of Nessler Reagent by following these instructions:

Standard Preparation

Use the following standards to make a calibration curve. See *Preparing a User-Entered Calibration Curve* on page 49, for more information and instructions. Prepare standards representing concentrations of 20, 60, 80, 100, 140 and 160 mg/L $\text{NH}_3\text{-N}$ as follows:

NITROGEN, TOTAL KJELDAHL, continued

- a) Using volumetric pipets, transfer 5.0, 15.0, 20.0, 25.0, 35.0, and 40.0 mL of 100 mg/L $\text{NH}_3\text{-N}$ standard solution into six separate 100-mL volumetric flasks. Dilute to volume with deionized water, stopper, and invert to mix.
- b) Begin at step 4 of the procedure using a 3-mL aliquot for the sample volume. Also prepare a blank solution by substituting a 3 mL aliquot of deionized water for sample in Step 4.

Note: Standard solutions are prepared as if a 25-mL volume was used for the digestion. Actual concentrations prepared in Step 1 are 5, 15, 20, 25, 35, and 40 mg/L $\text{NH}_3\text{-N}$. These represent original concentrations of 20, 60, 80, 100, 140, and 160 mg/L $\text{NH}_3\text{-N}$, based on the 25 to 100 mL dilution in the digestion.

User Entered Calibration Settings For TKN

Program # = 101 to 105

Wavelength = 420 nm

Resolution = 0 mg/L

Method Performance

Precision

In a single laboratory using a standard solution of 64 mg/L TKN and two representative lots of reagent with the instrument, a single operator obtained a standard deviation of ± 1.0 mg/L TKN.

Estimated Detection Limit

The estimated detection limit for program 65 is 2 mg/L TKN. For more information on the estimated detection limit, see *Section 1*.

Summary of Method

“Total Kjeldahl Nitrogen” (also called crude protein) refers to the combination of ammonia and organic nitrogen. Organically-bound in the trinegative state, it is converted into ammonium salts by the action of sulfuric acid and hydrogen peroxide. The ammonia is then analyzed by a modified nessler method test. The Mineral Stabilizer complexes calcium and magnesium. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color forms, proportional to the ammonia concentration.

NITROGEN, TOTAL KJELDAHL, continued

Pollution Prevention And Waste Management

Nessler reagent contains mercuric iodide. Both the sample and blank will contain mercury (D009) at concentrations regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. See Section 3 for more information on proper disposal of these materials.

REQUIRED REAGENTS

Total Kjeldahl Nitrogen Reagent Set 24953-00

Includes: (1) 21196-49, (1) 23766-26, (1) 21194-49, (1) 23765-26, (1) 282-32H,
(1) 23144-26, (1) 979-49, (1) 22519-26

Description	Quantity Required		Cat. No.
	Per Test	Unit	
Hydrogen Peroxide, 50%	20 mL.....	490 mL.....	21196-49
Mineral Stabilizer	6 drops.....	50 mL SCDB.....	23766-26
Nesslers Reagent.....	2 mL.....	500 mL.....	21194-49
Polyvinyl Alcohol Dispersing Agent.....	6 drops.....	50 mL SCDB.....	23765-26
Potassium Hydroxide Standard Solution, 8.0 N	varies	100 mL MDB.....	282-32H
Potassium Hydroxide Standard Solution, 1.0 N	varies	50 mL SCDB.....	23144-26
Sulfuric Acid, ACS.....	6 mL.....	500 mL.....	979-49
TKN Indicator Solution	2 drops.....	50 mL SCDB.....	22519-26
Water, deionized.....	varies	4 L.....	272-56

REQUIRED APPARATUS

Boiling Chips, silicon carbide.....	2-3	500 g.....	20557-34
Cylinder, graduated, mixing, tall-form, 25 mL.....	2	each.....	20886-40
Pipet, TenSette, 0.1 to 1.0 mL.....	1	each.....	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	2.....	50/pkg.....	21856-96
Safety Shield, for Digesdahl	1	each.....	50040-00
Sample Cell, 10-20-25 mL, w/ cap.....	2	6/pkg.....	24019-06

Select one based on available voltage:

Digesdahl Digestion Apparatus, 115 V	1	each.....	23130-20
Digesdahl Digestion Apparatus, 230 V	1	each.....	23130-21

OPTIONAL REAGENTS

Ammonia Nitrogen Standard Solution, 1 mg/L NH ₃ -N.....	500 mL.....	1891-49
Ammonia Nitrogen Standard Solution, Voluette Ampule, 150 mg/L NH ₃ -N, 10 mL	16/pkg.....	21284-10
Ammonia Nitrogen Standard Solution, 100 mg/L NH ₃ -N.....	500 mL.....	24065-49
Nitrogen Standard, Primary	3/set.....	22778-00

NITROGEN, TOTAL KJELDAHL, continued

OPTIONAL APPARATUS

Description	Unit	Cat. No.
Ampule Breaker Kit	each	21968-00
Balance, AccuLab Pocket Pro 250B	each	27969-00
Bottle, glass dispenser, 118 mL.....	each	591-00
Bottle, plastic wash, 1000 mL.....	each	620-16
Cylinder, graduated, 50 mL.....	each	508-41
Flask, volumetric, 100 mL, Class A.....	each	14574-42
Mini Grinder, 120 V.....	each	20991-00
pH Paper, 1 to 11 pH units	5 rolls/pkg	391-33
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28
Pipet, volumetric, Class A, 0.50 mL	each	14515-34
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Pipet, volumetric, Class A, 2.00 mL	each	14515-36
Pipet, volumetric, Class A, 5.00 mL	each	14515-37
Pipet, volumetric, Class A, 10.00 mL	each	14515-38
Pipet, volumetric, Class A, 15.00 mL	each	14515-39
Pipet, volumetric, Class A, 20.00 mL	each	14515-20
Pipet, volumetric, Class A, 25.00 mL	each	14515-40
Safety Glasses	each	18421-00

For Technical Assistance, Price and Ordering

In the U.S.A.—Call 800-227-4224

Outside the U.S.A.—Contact the Hach office or distributor serving you.